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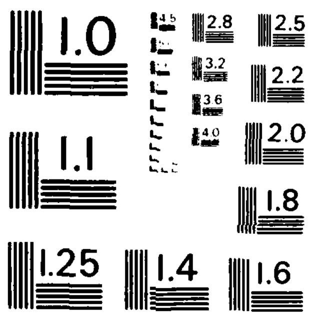

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

A study was conducted to determine the effects of crystallographic orientation and rhenium content on the creep behavior of a single crystal nickel-base superalloy. Creep tests conducted at 982°C (1800°F) and 220 MPa (32 ksi) on Alloy 444 single crystals show that there is an increase in creep strength as the specimen axis is varied from [001] to 26° from [001]. This orientation effect is favorable for use of nickel-base superalloy single crystal as turbine blade materials Creep deformation occurs by slip on {111} <110 > systems. The rate determining step appears to the shear of gamma prime (γ') precipitates by

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Deformation is relatively homogeneous with little if any lattice rotation observed. Preferential coarsening of the γ' particles perpendicular to the [001] component of the stress axis is observed. This results in the γ' particles becoming flattened pancake-shaped particles. This preferential coarsening occurs after as little as 30 hours in second stage creep.

Substitution of rhenium for tungsten in Alloy 444 increases creep strength. The mechanism for this effect is the subject of a continuing investigation.

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INTERIM REPORT

DEFORMATION AND FRACTURE OF ADVANCED ANISOTROPIC SUPERALLOYS

by

D. N. Duhl, M. L. Gell, A. F. Giamei and G. R. Leverant

PRATT & WHITNEY AIRCRAFT GROUP
UNITED TECHNOLOGIES CORPORATION
EAST HARTFORD, CONNECTICUT

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DR. ALAN H. ROSENSTEIN, PROGRAM MANAGER
AFOSR/NE - Bldg 410
Bolling AFB, Wash 20332

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1.0 ABSTRACT

A study was conducted to determine the effects of crystallographic orientation and rhenium content on the creep behavior of a single crystal nickel-base superalloy. Creep tests conducted at 982°C (1800°F) and 220 MPa (32 ksi) on Alloy 444 single crystals show that there is an increase in creep strength as the specimen axis is varied from [001] to 26° from [001]. This orientation effect is favorable for use of nickel-base superalloy single crystal as turbine blade materials. Creep deformation occurs by slip on {111} $<110>$ systems. The rate determining step appears to be the shear of gamma prime (γ') precipitates by $\frac{a}{2} <110>$ dislocations.

Deformation is relatively homogeneous with little if any lattice rotation observed. Preferential coarsening of the γ' particles perpendicular to the [001] component of the stress axis is observed. This results in the γ' particles becoming flattened pancake-shaped particles. This preferential coarsening occurs after as little as 30 hours in second stage creep.

Substitution of rhenium for tungsten in Alloy 444 increases creep strength. The mechanism for this effect is the subject of a continuing investigation.

2.0 INTRODUCTION AND SUMMARY OF SIGNIFICANT RESULTS

2.1 Introduction

The trend in the development of creep resistant wrought and cast alloys for gas turbine applications has been and continues to be in the direction of aligned microstructures. Oxide dispersion strengthened alloys, directionally solidified superalloys and directionally solidified eutectics all have microstructures which are aligned to provide maximum creep properties in the direction of principal component loading. The mechanical properties of these materials in directions not parallel to the aligned microstructure, however, are generally lower. That is, the properties are anisotropic.

2.2 First Year Effort

In the first year's effort of this program, under Air Force Contract F44620-76-C-0028, the anisotropy of tensile and compressive properties was studied for the $\gamma/\gamma'+\delta$ (Ni-20wt/pctCb-2.5wt/pctAl-6.0wt/pctCr) directionally solidified eutectic alloy. This eutectic system is under evaluation for turbine blade applications in advanced gas turbine engines.

Tensile and compression specimens tested at temperatures below the softening point of the δ (Ni_3Cb) reinforcing phase (~ 1050°K) were examined using optical and transmission microscopy to investigate the orientation dependence of deformation and fracture modes. In this temperature range, there is a large difference between longitudinal and transverse tensile ductility. Tension tests parallel to the freezing direction exhibit ductilities in excess of five percent while those perpendicular to the freezing directions exhibit ductilities less than one percent. Metallographic examination of broken transverse tensile specimens shows no preferred failure mode, such as interfacial delamination, which would account for this limited transverse ductility.

Analysis of the orientation dependence for operation of the known $\{211\}$ and $\{011\}$ twinning and (010) slip deformation modes in the Ni_3Cb phase indicates that $\{211\}$ twinning (four independent systems) which occurs in longitudinal tensile loading and provides the eutectic with adequate longitudinal ductility at all temperatures cannot operate on transverse tensile loading because either the twinning strains are negative or the Schmid factors are very low ($<$ 0.013) in the limited range of transverse orientations where $\{211\}$ twin strains are positive. Examination of tested specimens using light and transmission microscopy confirms the predicted orientation dependence of the Ni_3Cb deformation modes. This is summarized in Table I.

TABLE I

ORIENTATION DEPENDENCE OF TWINNING IN Ni_3Cb PHASE OF DIRECTIONALLY SOLIDIFIED $\gamma/\gamma'-\delta$

$\gamma/\gamma'-\delta$ Loading	Observed Twinning Planes	Number of Independent Twinning Systems
Longitudinal Tension	{211}	4
Longitudinal Compression	None (Perfect Alignment) 011 (Slight Misalignment)	None
Transverse Tension	{011}	2
Transverse Compression	{211} {011}*	6

*Predominant system

In particular, {211} twins are absent in transverse tensile specimens, with only limited {011} twinning (two systems) being found in selected grains. This leads to the conclusion that the relatively low transverse tensile ductility of the eutectic results from the limited number of deformation systems which operate in the Ni_3Cb reinforcing phase below the softening temperature.

2.3 Second Year Effort

2.3.1 Effect of Crystallographic Orientation on 982°C (1800°F) Creep Properties of Single Crystal Superalloys

Leverant et al.[1,2] have investigated the orientation dependence of creep behavior in nickel-base superalloy single crystals at 760°C (1400°F) and 857°C (1575°F). At 760°C, which is representative of turbine blade root attachment temperatures, creep deformation was found to be highly orientation dependent because of planar deformation occurring on {111} < 112 > slip systems. At 857°C, which is lower than creep-controlling temperatures in many blades, deformation was found to be more homogeneous, less orientation dependent and occurring on {111} < 110 > slip systems. In this investigation, the orientation dependence of creep was studied at 923°C (1800°F), a temperature representative of blade operating temperatures in current and advanced engines.

The single crystal superalloy selected for this work is designated Alloy 444. The alloy has been based on the high strength nickel-base superalloy MAR-M 200 with the grain boundary strengthening elements, boron, carbon, hafnium and zirconium, absent from the composition. Table II gives the composition of the Alloy 444 single crystal material employed in this work together with the nominal MAR-M 200 + Hf composition.

TABLE II

ALLOY COMPOSITION (WEIGHT PERCENT)

	Al	Ti	Cb	Cr	W	Co	Hf.	C	B	Zr	Ni
Alloy 444	5.1	1.98	0.91	8.6	11.1	---	---	---	---	---	Balance
MAR-M 200 + Hf	5.0	2.00	1.00	9.0	12.0	10.0	1.8	0.15	0.015	0.05	Balance

Creep specimens were machined from heat treated (1288°C/4 hours + 1079°C/4 hours + 871°C/32 hours) single crystal Alloy 444 bar castings. The crystallographic orientation of each bar is determined by the Laue back reflection technique and redetermined on each machined specimen to confirm the precise orientation of the tensile axis. All specimens were creep tested at 982°C in constant load creep machines under a load of 220 MPa. The results of the creep tests are listed in Table III and the orientation of the specimens is shown in Figure 1.

TABLE III

CREEP TEST RESULTS OF ALLOY 444 SINGLE CRYSTALS
(982°C/220 MPa - 1800°F/32 KSI)

Specimen Number	Orientation of Specimen Axis (Degrees from [001])	Primary Creep Strain (%)	Minimum Creep Rate $\times 10^4$ (Hours)	Time to 1% Creep (Hours)	Rupture Life (Hours)	Failure Elongation (%)
1	5.5	0.3	2.33	35.6	118.9	16.9
2	10.0	0.25	1.62	52.3	154.7	21.6
3	12.0	0.2	1.69	53.7	142.2	16.6
4	12.0	0.6	0.738	76.6	188.6	22.1
5	6.5	0.25	1.88	47.4	144.1	8.2
6	13.0	0.3	0.922	92.3	217.3	13.3
7	5.0	0.32	1.41	Discontinued at 0.9%/150 hours.		
8	8.5	0.35	1.28	62.8	193.7	13.3
9	10.0	0.15	1.66	49.8	156.8	18.0
10	5.0	0.5	1.45	51.7	Discontinued at 1.1%/ 58.1 hours.	
11	5.0	0.2	1.65	45.2	167.2	22.5
12	5.5	Discontinued at 0.2%/19.8 hours.				
14	10.5	0.2	1.36	65.4	190.8	22.4
16	7.0	0.3	1.61	52.4	161.2	19.2
17	7.5	0.2	2.05	41.6	115.7	19.2
18	26.0	---	1.33	Discontinued at 0.81%/59.7 hours.		
19	7.0	0.25	1.44	64.0	202.8	16.1
20	22.5	0.1	1.19	78.9	153.8	
21	25.0	0.1	1.34	69.4	130.1	14.6
22	18.5	0.25	0.954	84.9	212.9	18.9
23	11.0	0.2	1.54	55.8	185.2	14.2
24	7.0	0.35	1.81	44.8	Discontinued at 4.7%/ 124.6 hours.	

TABLE III

- continued -

Specimen Number	Orientation of Specimen Axis (Degrees from [001])	Primary Creep Strain (%)	Minimum Creep Rate x 10^4 (Hours)	Time to 1% Creep (Hours)	Rupture Life (Hours)	Failure Elongation (%)
25	0	Discontinued at 0.14%/13.9 hours.				
26	0	0.15	2.65	30.7	Discontinued at 1.0%/ 30.7 hours.	
27	1.5	0.15	1.72	52.5	125.5	11.3
28	6.0	0.2	1.80	44.7	112.7	18.9
29	1.0	0.2	1.63	47.1	134.2	22.2

The creep data show an effect of orientation on both minimum creep rate (MCR) and time to 1% creep, with creep resistance increasing as orientations move away from the [001] direction. The effect of MCR on orientation is plotted in Figure 2. Statistical analysis of the data indicates that the correlation factor between MCR and orientation is 0.57 (correlation of 0.67 was found between time to 1% creep and orientation), where a factor above 0.75 indicates a significant effect. [redacted]57°C, [redacted] the [001] - [redacted] [001] - [110] portion of the standard stereographic triangle. The extent of primary creep at 982°C is very limited as was found for creep at 857°C and no incubation period was observed.

These results are significant with respect to the potential application of single crystal nickel-base superalloys as turbine blade materials. They indicate that the blade axes can [redacted]

Transmission electron microscopy was employed to define the active slip systems. Using a two beam method, the active slip systems were identified as {111} < 110 >, for a [001] oriented specimen. Similar results were found for creep at 857°C. Comparison of the dislocation substructure in primary creep (0.144% ϵ) at 982°C (Figure 3) with that previously reported at 760 and 857°C (Figure 5, Reference 1) shows the similarity in dislocation substructure during creep at 982 and 857°C. The absence of stacking faults during creep at 982°C can be seen in Figure 3. This is consistent with < 110 > slip as opposed to < 112 > slip which requires the formation of stacking faults. Paired dislocations together with the concentration of dislocations at the γ/γ' interface and absence of dislocation in the γ' particles is also consistent with shearing of the γ' particles by pairs of $a/2 < 110 >$ dislocations being the rate-determining step, as was found for creep at 857°C.

Analysis of lattice rotation of the failed specimens showed very little rotation (< 4°) indicating that slip is occurring on several systems. At 982°C, cross-slip is probably obscuring any effects of lattice rotation.

During creep deformation, the γ' particles coarsen with growth occurring perpendicular to the stress axis for tensile loading as first shown by Tien and coworkers[3-5]. Figure 4 shows the three {100} faces of a specimen 26° from [001] deformed at 982°C. The γ' particle coarsening occurs perpendicular to the [001] direction, and not perpendicular to the stress axis. Comparison of Figure 5 with Figure 4b shows that stress is needed to cause the preferential growth of the γ' particles. Figure 6 shows that after as little as fourteen hours or 0.14% strain (primary second stage creep), the γ' particles are fully coarsened.

2.3.2 <u>Role of Rhenium on Creep Deformation of Superalloys at 982°C</u>

Three alloys were prepared with two, four and six weight percent rhenium replacing an equivalent amount of tungsten in Alloy 444. As tungsten and rhenium have similar atomic weights, 183.85 and 186.2 respectively, substitution on a weight percent basis is nearly equivalent to substitution on an atomic percent basis. The compositions of the three rhenium-modified alloys are listed in Table IV.

TABLE IV

COMPOSITION OF RHENIUM MODIFIED ALLOYS (WT %)

Alloy	Al	Ti	Cb	Cr	W	Re	Ni
50	5.0	1.87	0.95	8.8	9.2	2.2	balance
60	5.1	1.87	0.95	8.7	7.2	3.8	balance
70	5.0	1.87	0.95	8.6	5.5	6.4	balance

The crystallographic orientation of the rhenium-modified alloys are shown in Figure 7 and the 982°C/220 MPa creep data are summarized in Table V. Substitution of rhenium for tungsten increases creep strength as shown in Figure 8 where time to 1% creep is plotted as a function of weight percent rhenium.

TABLE V

CREEP RESULTS OF RHENIUM-MODIFIED ALLOYS AT 982°C/220 MPA (1800°F/32 KSI)

Specimen Number	Primary Creep Strain (%)	Minimum Creep Rate x 10^4 (Hours)	Time to 1% Creep (Hours)	Rupture Life (Hours)	Failure Elongation (%)
53	0.45	0.764	84.1	268.1	31.6
54	0.55	0.579	122.6	318.4	28.7
56	0.8	0.503	91.5	376.2	28.6
61	0.5	0.897	98.7	273.7	31.1
65	0.5	0.860	82.4	245.0	31.7
71	0.4	0.559	149.5	322.4	25.6
72	0.45	0.383	184.8	339.3	19.4

REFERENCES

1. G.R. Leverent, B.H. Kear, and J,M. Oblak, Met. Trans. Vol. 4 (1973),p.355.
2. G.R. Leverant and B.H. Kear, Met. Trans., Vol.1 (1970).p.491.
3. J.K. Tien and S.M. Copley, Met. Trans., Vol. 2 (1971),p.215.
4. J.K. Tien and S.M. Copley, Met. Trans., Vol. 2 (1971),p.543.
5. J.K. Tien and R.P. Gamble, Met. Trans., Vol. 3 (1972),p.2157.

3.0 PUBLICATIONS AND PRESENTATIONS FROM AFOSR SPONSORED WORK

- A presentation of progress to date was made to I. Machlin's joint DoD/NASA Committee on DS Eutectics at NAV AIR in Washington, DC on 4 March 1976.
- A presentation entitled, "The Anisotropy of Deformation and Fracture in a Directionally Solidified Ni/Ni_3Al-Ni_3Cb Lamellar Eutectic Alloy", was made at the Fall Meeting of TMS-AIME, Niagara Falls, NY in September 1976.
- A paper entitled "the Anisotropy of Deformation and Fracture in a Directionally-Solidified Ni/Ni_3Al-Ni_3Cb Lamellar Eutectic Alloy" was published in Metallurgical Transactions, Vol. 8A,No.1.pp.83-89, January 1977.
- A presentation will be made at the Fall Meeting of TMS-AIME, St. Louis, Missouri, in September 1978.

4.0 PERSONNEL INVOLVED IN PROGRAM

I. FIRST YEAR

G.R. Leverant
K.D. Sheffler
A. Yuen
R.H. Barkalow

II. SECOND YEAR

D.N. Duhl
M.L. Gell
A.F. Giamei
G.R. Leverant

III. THIRD YEAR

A.F. Giamei

5.0 COUPLING ACTIVITIES WITH AIR FORCE PROGRAMS AND IR&D EFFORTS

The AFOSR sponsored work in this program on deformation and fracture of advanced anisotropic turbine airfoil materials, $\gamma/\gamma'-\delta$ eutectic and single crystal superalloys have been and continue to be materials receiving a great deal of IR&D support at Pratt & Whitney Aircraft. DS eutectics have been an area of interest to the Air Force as evidenced by the AFML program activity in this area. Single crystal nickel-base superalloys are specififed for advanced versions of the F100-PW-100 engine that powers the F15 and F16 aircraft and for the advanced technology demonstrator ATEGG/JTDE.

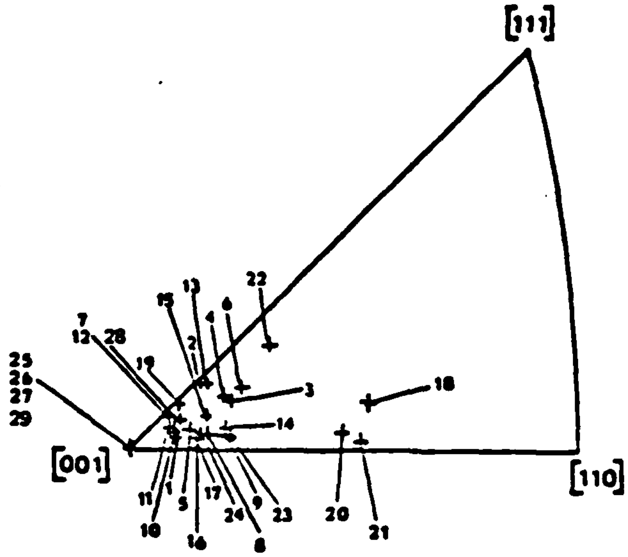


FIGURE 1. ORIENTATION OF SPECIMEN AXIS

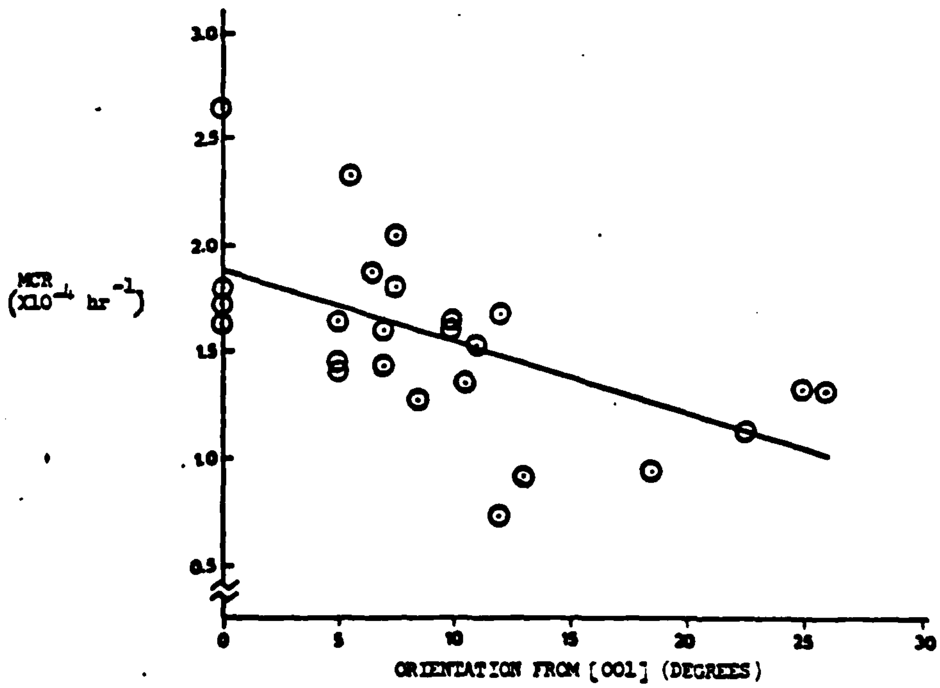


FIGURE 2. EFFECT OF MINIMUM CREEP RATE (MCR) ON ORIENTATION FOR CREEP AT 982°C/220 MPa (1800°F/32 ksi)

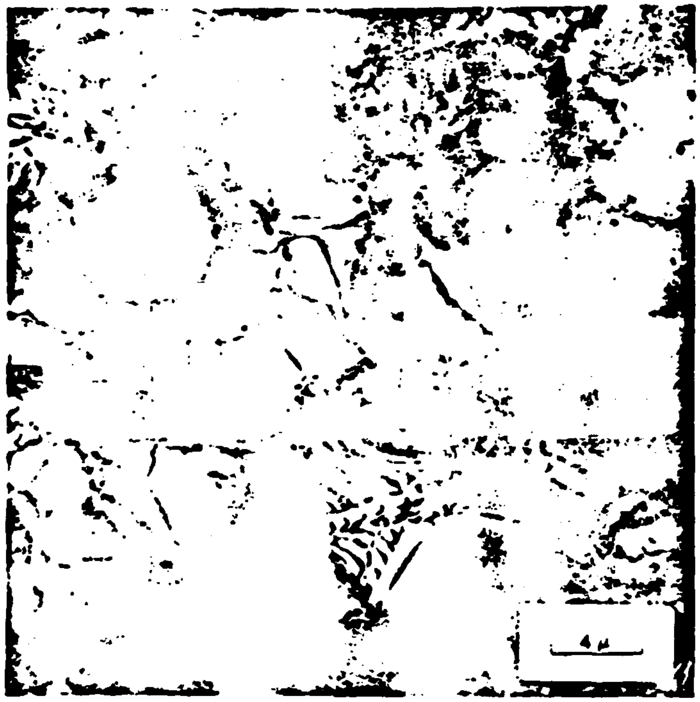


FIGURE 3. DISLOCATION STRUCTURE IN PRIMARY CREEP (0.144% ε) AT 982°C/220 MPa (1800°F/32 ksi)

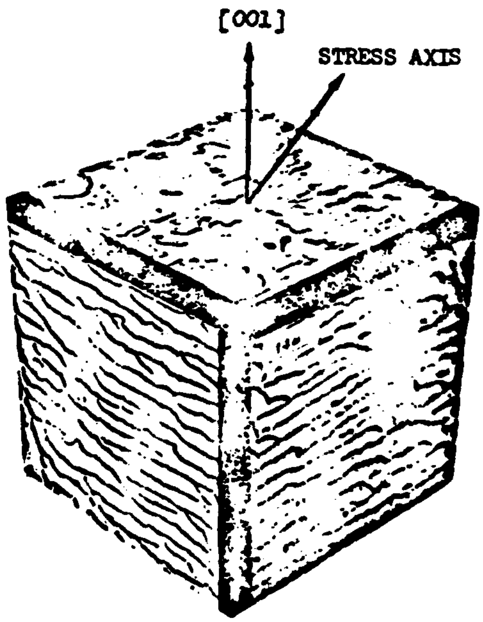


FIGURE 4. γ' COARSENING ON {100} PLANES AFTER CREEP AT 982°C/220 MPa (1800°F/32 ksi)

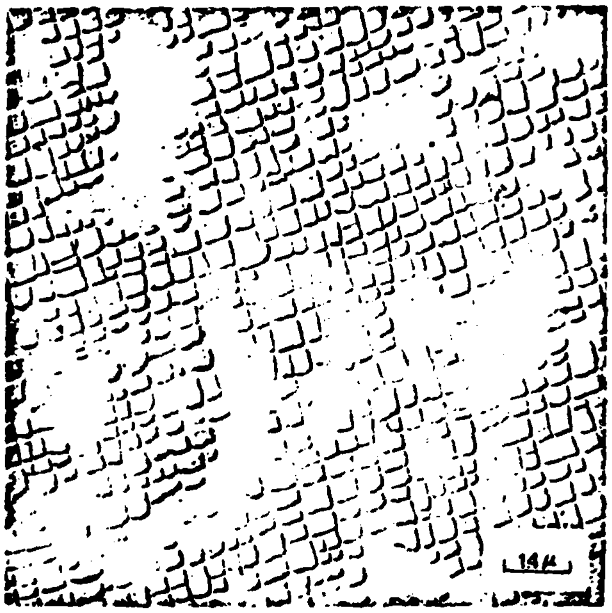
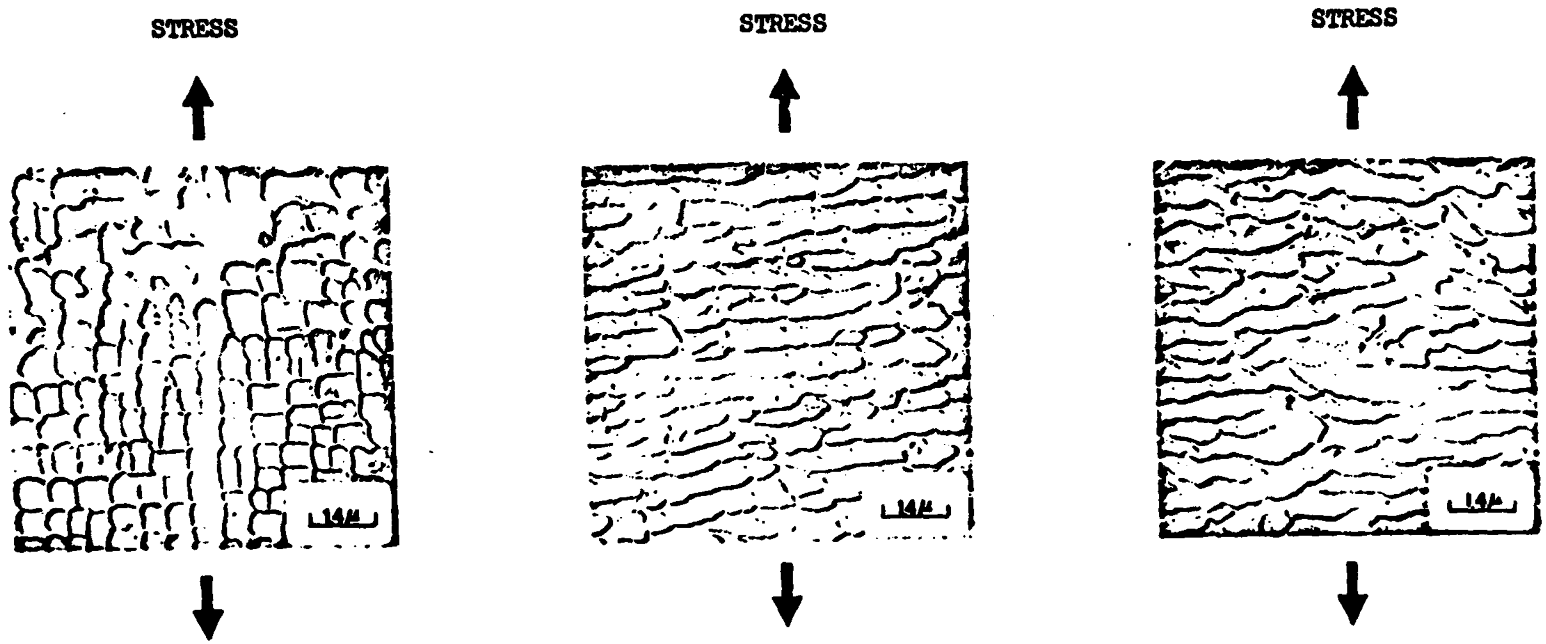


FIGURE 5. EFFECT OF ANNEALING (NO STRESS) FOR 200 HOURS AT 982°C (1800°F) ON γ' PARTICLE COARSENING



(010) PLANE 0.144%ε PRIMARY 14hr?

(010) PLANE 1.0% ε SECONDARY 39,111

(010) PLANE 125.5 HOURS FAILED

FIGURE 6. EFFECT OF CREEP AT 982°C/220 MPa (1800°F/32 ksi) on γ' PARTICLE COARSENING

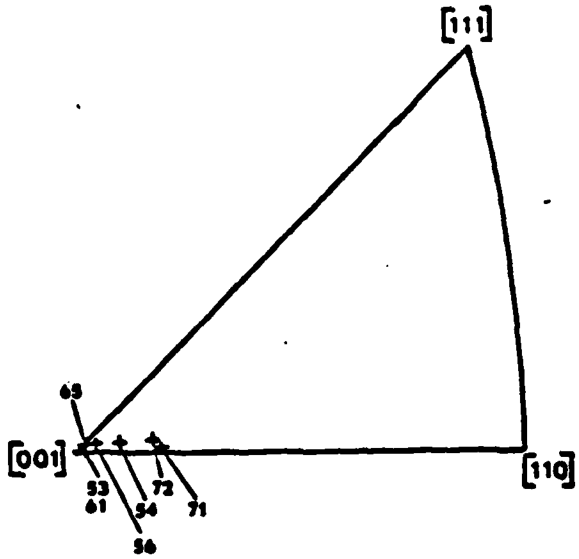


FIGURE 7. ORIENTATION OF RHENIUM MODIFIED ALLOYS

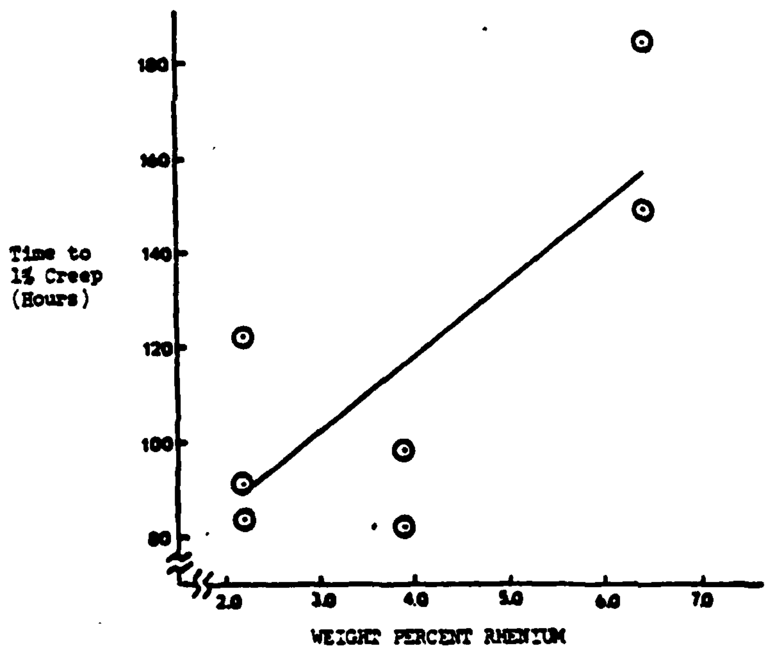


FIGURE 8. EFFECT OF RHENIUM CONTENT ON 982°C/220 MPa (1800°F/32 ksi) CREEP RESISTANCE

... to the shear of gamma prime (γ') precipitates by ... <110> systems. The rate

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